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- 1 5890 Hewlett-Packard GC and Tekmar LSC 2 single position sample concentrator equipped with dual FID detectors and connected to a linear chart recorder for sample screening.
- 1 Hewlett-Packard 7974 Magnetic Tape Drive for RTE 6/1000 data archival. Two locked cabinets for tape storage. UNIX system is stand alone.
- RTE 6/1000 Rev. E. and UNIX Series 400 software and peripheral hardware.

6.4 Miscellaneous

- assorted syringes (10, 25, 50, 100, 500 and 1000 uL)
- 5 mL and 25 mL luer-lock gas-tight syringes
- assorted purge vessels (water/soil, 5/25 mL, 5 g)
- top-loading balance, capable of weighing to ± 0.1 g, stainless steel spatula
- assorted amber and clear teflon-lined screw-capped vials (1.5 - 2.0 mL, 3.5 - 5.0 mL)
- cleaned 40 mL vials w/teflon-lined screw-caps

7.0 PREVENTIVE MAINTENANCE

Instrumental maintenance can be categorized as daily, yearly and "as required".

7.1 Daily Maintenance

The most routinely performed maintenance includes:

- position rinse
- tube baking after sample analysis
- oven bake after high level samples

7.2 Yearly Maintenance

The only scheduled yearly maintenance includes changing the pump oil. This is done by an HP representative.

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7.3 "As Required"

Most maintenance is done on an "as needed" basis, is operator determined and can be categorized as GC, Tekmar, or MS related.

7.3.1 GC Related

- change column; condition new column
- clean separator; change separator
- check helium flow rate
- change gas cylinders and moisture trap

7.3.2 MS Related

- clean source/rods and anything associated with that activity

7.3.3 Tekmar Related

- rinse positions
- change positions; change parts of positions
- change transfer line; clean transfer line
- replace trap; condition new trap
- refurbish Tekmar
- check purge pressure and flow rate

Each required maintenance may be performed for a variety of reasons. Certain trouble-flags will indicate what maintenance procedures may be required. Refer to the GC/MS Maintenance Manual. An example appears in Figure 1. The entry number is then transferred to the appropriate operation logbook.

8.0 STANDARDS AND QC SOLUTIONS

- 8.1** All neat standards/kits received are recorded in the Neat Standards Logbook. See Figure 2. A code is written on the bottle/kit and recorded in the log book. All neat standards are then stored in a separate freezer at approximately -10°C until needed. See logbook for examples. The standard is issued a Neat Standards Reference Number which is used to track all standards as they are used as is or in preparation of stock/working solutions.

8.2 Surrogate Spiking Solution

Stock surrogates are purchased as solutions from Supelco. The following surrogates are used:

Compound	Concentration	Catalog No.
4-Bromofluorobenzene	2000 ug/mL	4-8083
1,2-Dichloroethane-d ₄	2000 ug/mL	4-8941
Toluene-d ₈	2000 ug/mL	4-8087

These are stored in a separate freezer at approximately -10°C in the dark and kept for a period of one year. The working solution is prepared with internal standard solution. See next section.

8.3 Internal Standard Spiking Solutions

Stock internal standards are purchased as solutions from Supelco. The following internal standards are used:

Compound	Concentration	Catalog No.
Pentafluorobenzene	2000 ug/mL	4-8945
Chlorobenzene-d ₅	2000 ug/mL	4-8086
1,4-Difluorobenzene	2000 ug/mL	4-8944
1,2-Dichlorobenzene-d ₄	2000 ug/mL	4-8952

These are stored at -10°C in the dark. Unopened ampules are kept for a period of one year. Opened ampules are kept for a period of 6 months or until used. The contents of the opened ampules is transferred to a 1.5 - 2.0 mL amber teflon-lined screw-capped vial. The WORKING internal standard/surrogate solution is prepared as follows:

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Compound	Volume (uL)	MeOH (mL)	Concentration
Pentafluorobenzene	50	2 mL	50 ppm
Chlorobenzene-d ₅	50		
1,4-Difluorobenzene	50		
1,2-Dichlorobenzene-d ₄	50		
Bromofluorobenzene	50		
1,2-Dichloroethane-d ₄	50		
Toluene-d ₈	50		

This is stored in a 3.5 - 5.0 mL amber teflon-lined screw-capped vial at approximately -10°C in the dark. All standard preparation is recorded in the Volatiles Standard Logbook. See Figure 3. Each standard is issued a Standard Reference Number (SRN). Working ISS/SSS is prepared every two weeks. Addition of 5 uLs to 5 mLs/25 mLs volume results in a concentration of 50/10 ppb (soil/water).

All standard labels contain the following information: standard description, concentration, date prepared, analyst and expiration date. All neat standards are returned to storage.

8.4

Stock Purgeable Standards

These are obtained as neat solutions from Supelco. The contents of each solution, catalog number and concentrations appear on the next page. The ampules are stored at approximately -10°C in the dark. Stock ampules are good for a period of one year. Upon opening, all contents are transferred to 1.5 - 2.0 mL amber, teflon-lined screw-capped vials. Opened stocks can be used for a period of three months. Listed are compounds in Method 8260 and includes those done on a regular basis. Other standards, if needed, are either purchased as neat solutions or neat standards from Supelco, Chem Service or other certified supplier.

See appropriate entries in the Neat Standards/VOA Standards Logbook. All neat standards are still logged into the Neat Standard Logbook.

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8.4.1 Working 5-Point Standard

Each stock calibration mix is transferred to a 1.5 - 2.0 mL amber teflon-lined screw-capped vial just prior to use. The following tables describes the preparation procedure:

Waters

Stock Compound/Mix	Volume (uL)	Vol (MeOH)	Conc.
2000 ppm VOC Mix 1	25	diluted to 2 mLs	25 ng/uL each component
2000 ppm VOC Mix 2	25		
2000 ppm VOC Mix 3	25		
2000 ppm VOC Mix 4	25		
2000 ppm VOC Mix 5	25		
2000 ppm VOC Mix 6	25		

Soils

Stock Compound/Mix	Volume (uL)	Vol (MeOH)	Conc.
2000 ppm VOC Mix 1	100	diluted to 2 mLs	100 ng/uL each component
2000 ppm VOC Mix 2	100		
2000 ppm VOC Mix 3	100		
2000 ppm VOC Mix 4	100		
2000 ppm VOC Mix 5	100		
2000 ppm VOC Mix 6	100		

NOTE: The other standards used in the calibration for the additional compounds are the same as those listed in the Method 8240 SOP. The concentration is adjusted for either the soil or water analysis. See the continuing calibration standard preparation.

This gives 2 mLs of solution which is stored in a 1.5 - 2.0 mLs amber teflon-lined screw-capped vial at -10°C in the dark. All standard preparation is recorded in the Volatiles Standard Logbook. The Working 5-Point Solution is prepared every two weeks. See above for label information.

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Stock Purgeables Supelprime™ - HC Calibration Standards

Volatile Organic Compounds Mix #1 Catalog No. 4-8775

This mixture contains 2000 ug/mL of each of the following components in methanol:

Chlorobenzene	4-Chlorotoluene
p-Xylene	tert-butylbenzene
o-Xylene	sec-Butylbenzene
Isopropylbenzene	1,3-Dichlorobenzene
n-Propylbenzene	1,4-Dichlorobenzene
2-Chlorotoluene	1,2-Dichlorobenzene

Volatile Organic Compounds Mix #2 Catalog No. 4-8777

This mixture contains 2000 ug/mL of each of the following components in methanol:

Benzene	Toluene
Bromobenzene	1,2,3-Trichlorobenzene
n-Butylbenzene	1,2,4-Trichlorobenzene
Ethylbenzene	1,2,4-Trimethylbenzene
p-Isopropyltoluene	1,3,5-Trimethylbenzene
Naphthalene	m-Xylene
Styrene	

Volatile Organic Compounds Mix #3 Catalog No. 4-8779

This mixture contains 2000 ug/mL of each of the following components in methanol:

1,2-Dibromo-3-chloropropane	Hexachlorobutadiene
1,2-Dibromoethane	1,1,1,2-Tetrachloroethane
1,2-Dichloroethane	1,1,2,2-Tetrachloroethane
1,2-Dichloropropane	1,1,2-Trichloroethane
1,3-Dichloropropane	Trichloroethylene
1,1-Dichloropropylene	1,2,3-Trichloropropane
1,3-Dichloropropylene*	

*Mixture of cis-1,3-Dichloropropylene and trans-1,3-Dichloropropylene, 4000 ug/mL total.

Volatile Organic Compounds Mix #4 Catalog No. 4-8786

This mixture contains 2000 ug/mL of each of the following components in methanol:

Bromochloromethane	1,1-Dichloroethane
Bromoform	2,2-Dichloropropane
Carbon tetrachloride	Tetrachloroethylene
Chloroform	1,1,1-Trichloroethane
Dibromomethane	

Volatile Organic Compounds Mix #5 Catalog No. 4-8797

This mixture contains 2000 ug/mL of each of the following components in methanol:

Bromodichloromethane	cis-1,2-Dichloroethylene
Dibromochloromethane	trans-1,2-Dichloroethylene
1,1-Dichloroethylene	Methylene Chloride

Volatile Organic Compounds Mix #6 Catalog No. 4-8799

This mixture contains 2000 ug/mL of each of the following components in methanol:

Bromomethane	Dichlorodifluoromethane
Chloroethane	Trichlorofluoromethane
Chloromethane	Vinyl Chloride

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The 5-point standards are prepared just prior to calibration. These are used once and are not stored. The following recipe is used in preparation. Each level is a separate solution in a gas-tight syringe.

Waters: 25-mL Purge Volume

Concentration (ppb)	Stock Water 5-pt. Std. (uL)	Working ISS/SSS (uL)	Water Vol. (mLs)
20	20	5	25
15	15	5	25
10	10	5	25
5	5	5	25
1	1	5	25

Soils: 5-mL Purge Volume

Concentration (ppb)	Stock Soil 5-pt. Std. (uL)	Working ISS/SSS (uL)	Water Vol. (mLs)
200	10	5	5
150	7.5	5	5
100	5	5	5
50	2.5	5	5
10	1	5	5

8.5

Stock Matrix Spike Solution

The matrix spike compounds are obtained as neat standards from Supelco. See Neat Standard Logbook for references. The following matrix spike compounds are used:

Benzene	1,1-Dichloroethene
Chlorobenzene	Toluene
Trichloroethene	

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These are stored at approximately -10°C in the dark prior to use. Neat standards are kept for a period of one year or manufacturers date. Upon opening, the contents are transferred to amber vials. The transfer is recorded in the Standard Preparation Logbook. The standard is assigned an SRN. Labeling information is as above.

8.5.1 The STOCK matrix spike solution is prepared as follows:

Compound	Weight	Vol. (MeOH)	Conc.
Benzene	100 mg	10 ml	10,000 ppm
Chlorobenzene	100 mg		
Trichloroethene	100 mg		
1,1-Dichloroethene	100 mg		
Toluene	100 mg		

This is stored in a 3.5 - 5.0 mL amber teflon-lined screw-capped vial at approximately -10°C in the dark. All standard preparation is recorded in the Volatile Standards Logbook. Matrix spike stock solutions are kept for a period of 6 months or until QC indicates a new solution is required. See above for label information.

8.5.2 The WORKING matrix spike solution is prepared as follows:

Compound	Volume	Vol. (MeOH)	Conc.
Stock Matrix Soln.	25 uL	10 mL	25 ppm

This solution is stored at approximately -10°C in several 1.5 -2.0 mL amber teflon-lined screw-capped vials. All standard preparation is recorded in the Volatile Standard Logbook. Working matrix spike solutions are used for a period of 3 months or until low recoveries of the matrix spike compounds indicate a new solution is needed. See above for label information.

Addition of 10 uL of this solution to a sample results in all spike compounds at 50 ppb or 10 ppb (soil/water).

8.5.3 QC Charting

The QC matrix spike is prepared the same way as the standard by a different analyst and is a separate solution. A BS and BSD is completed once a month.

8.5.4 Stock BFB Solution

The BFB standard is purchased as a neat solution from Supelco.

Compound	Catalog No.	Concentration
Bromofluorobenzene	4-8083M	2000 ppm

The standard is stored at approximately -10°C in the dark and kept for a period of 1 year until opening. Upon opening, the solution is transferred to a 1.5 - 2.0 mL vial and assigned an SRN.

Stock	Amount (uL)	MeOH (uL)	Concentration
BFB	25 uL	1975 uL	25 ppm

Addition of 2 uL/5 mLs results in a concentration of 25 ppb.

All preparation is recorded in the Standards Preparation Logbook. All labels are completed as above. The Working BFB Solutions are kept for a period of 6 months.

NOTE: Intermediate and Working Solutions are never assigned an expiration date exceeding the expiration date of the neat/stock standards/solutions.

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8.6 Reagents8.6.1 Reagent Water

Approximately two (2) liters of water is continuously purged with pre-purified nitrogen. The two jars are alternately used. The reagent water is routinely demonstrated to be interference-free. All compounds are less than their EQL or 5X EQL for specific compounds.

8.6.2 Methanol

Purge and Trap B&J Methanol interference-free. Each lot number of methanol is checked for contamination prior to laboratory use and is documented in the QA/QC department.

9.0 PROCEDURE

Before an instrument is used as a measuring device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration is documented.

9.1 Calibration

An initial 5-point calibration is completed:

- as needed-continuing calibration can not be met
- after a source cleaning and/or column change
- any time a major repair or change has occurred with the instrument that affects calibration

Confirm that the GC/MSD or bench-top is stable and equilibrated. If at all possible, allow the instrument to equilibrate overnight at all operating temperatures if the source/column has been cleaned/changed. Prior to beginning initial calibration it is a good idea to:

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- check the background of air/water levels and base ion by scanning for appropriate ions and also visually inspecting the spectrum scan for any other possible and undesirable background.

- recheck the multiplier settings, after a source is cleaned the EM can most often be dropped.

9.2 PFTBA Autotune or Manual Tune

The instrument is first tuned in one of two ways: autotune or manual tune. The ion abundances in the calibration gas are best monitored near the temperature of analysis of BFB. Monitoring at this temperature produces the most representative cal gas scan and therefore the best estimate of BFB response.

9.2.1 If an AUTOTUNE is to be done, continue below. If not, skip to step 4. An autotune is not run before every 5-point calibration. If the instrument has been down for any reason previously listed or major difficulties in manual tune are encountered, an autotune is performed. Autotunes are generally NOT performed when an existing 5-point is being met.

9.2.2 Enter MSCAL (or appropriate command) to begin the autotune program. Key masses are 69, 219 and 502. A good explanation of the 10 steps and any help needed for any of the screens can be found in the Hewlett-Packard RTE-6/VM Data System Manual.

9.2.3 Follow instructions and retrieve a hardcopy of the autotune results. Check the following:

- passed/fail: in itself, not necessarily an indication of MS performance
- repeller and ion focus settings
- electron multiplier voltage

The repeller and EM voltages are good indicators of the sources' cleanliness. Generally, the lower the setting the cleaner the source. Other factors may however, supersede (i.e. the age of the multiplier) and a clean source will not always autotune these low. The EM is set by autotune program to produce an abundance for mass 69 around 15000. The operator can plan on having to increase this by 100-200 to achieve normal analysis sensitivity.

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Observe peak shape, absence of lead-ons/tailing, the resolution between isotopes, peak width and mass axis. A hardcopy of the profile scan is desirable, and can be filed with the autotune results.

9.2.4 If an AUTOTUNE has just been performed, continue here. If not, skip to step 7. Enter **MANUAL TUNE** and read the autotune (which was automatically stored in a file). For volatiles, edit the scan parameters to monitor ions 69, 131 and 219.

9.2.5 Enter one of several methods available and adjust the parameters (usually the ion focus, entrance lens and amu gain) to achieve the following relative abundances:

Mass	Relative Abundance
69	100%
131	32-40%
219	35-45%

These will vary with the MS. Mass 219 is usually 5-9% greater than mass 131. Adjust the EM voltage up 100 volts. If necessary, adjust the amu gain for peak shape and high-end isotope resolution. An overall peak-width of 0.500 is desirable.

Again, these adjustments and relative abundances may not guarantee that BFB will meet requirements, but is a good place to start.

9.2.6 Hardcopy the profile scan. This should be filed with the autotune results. This file can serve as a diagnostic tool and can also provide a starting point in the event the operator has trouble meeting the 5-point calibration.

NOTE: In volatiles, as opposed to semi-volatiles, there are a few more things that will affect continuing calibration. Although there may be times when adjusting the tune parameters may help, it will most likely be another problem (i.e. age of standards).

Save the changes to the appropriate Tune File. Exit the program.

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9.2.7 If an AUTOTUNE has not been performed, enter MANUAL TUNE and adjust any parameters, if need be. Adjustment may not be necessary, and not desirable, if problems in tuning or meeting the initial calibration have not been encountered. Hardcopy a profile scan and exit.

NOTE: Again, this step in volatiles is not as critical as it is in semi-volatiles. It may be desirable if the operator has been having problems with the instrument, but this is not always necessary. Observing and hardcopying the profile scan does serve as a reference for the next day.

9.3 BFB Analysis

Once the instrument is tuned, a 25 ng injection of 4-Bromofluorobenzene must meet criteria. The BFB can be purged or directly injected. The mass spectrum must meet the following criteria:

Mass	Ion Abundance
50	15-40% of mass 95
75	30-60% of mass 95
95	Base Peak, 100% rel. abund.
96	<2% of mass 95
173	<2% of mass 174
174	>50% of mass 95
175	5 - 9% of mass 174
176	>95% but <101% of mass 174
177	5 - 9% of mass 176

The BFB is analyzed by one of the methods in Appendix A. (These are examples, as methods may be adjusted.) Typical Tekmar conditions also appear in Appendix A. The EM voltage should be 100-200 volts above autotune. The abundances of the designated masses above **MUST** meet the criteria before analyses can begin. If necessary, enter MANUAL TUNE and adjust parameters. The instrument is tuned about every 12 hours of analysis.

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 9.4 Initial Calibration

Each 5-point calibration standard is analyzed according to one of the methods in Appendix A. These are examples. The EM voltage should be 100-200 volts above autotune.

Allow standards to come to ambient temperature.

Fill five 5 mL or 25 mL luer-lock gas-tight syringes with reagent water to overflowing. Replace the plunger and invert. Adjust to 5 mLs (or 25 mLs), confirming the absence of any air bubbles. Pull back slightly on the plunger to allow addition of standards.

Use the following as a guide:

Waters: 25-mL Purge Volume

Concentration (ppb)	Stock Water 5-pt. Std. (uL)	Working ISS/SSS (uL)	Water Vol. (mLs)
20	20	5	25
15	15	5	25
10	10	5	25
5	5	5	25
1	1	5	25

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Soils: 5-mL Purge Volume

Concentration (ppb)	Stock Soil 5-pt. Std. (uL)	Working ISS/SSS (uL)	Water Vol. (mLs)
200	10	5	5
150	7.5	5	5
100	5	5	5
50	2.5	5	5
10	1	5	5

The same tables appear in the standard section. Immediately add the standards to a clean and baked purge vessel. Following the parameters in Appendix A, analyze the 50 ppb or 10 ppb standards. A normal standard will appear very similar to the one in Figure 4. Quantitate the standard against the appropriate ID file. A short list example of one ID file appears in Appendix B. Note the area of the first internal standard. Too low an area will almost guarantee poor/unsatisfactory responses of low-response compounds and too high an area will result in saturation of some compounds at higher levels, resulting in false low response factors at high concentrations.

Response factors are calculated by the data system as follows:

$$RF = \frac{A_x \times Q_s}{A_s \times Q_x}$$

Where:

- A_x = ion abundance for analyte
- A_s = ion abundance for its internal standard
- Q_x = concentration of its internal standard
- Q_s = concentration of analyte

(Response Factors have no units)

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The appropriate quant ion must be in the ID file. See an example of a full-listed ID file in Appendix B. A listing of the target compounds with their appropriate internal standards also appears in Appendix B. Confirm the presence of all targets and the separation of non- co-eluting compounds. Note the response factors for the gasses. If necessary, prepare new standards.

If adjustments to the acquisition parameters are necessary, make them and re-analyze the 50 ppb or 10 ppb standard.

After a good standard is analyzed it is desirable to update the RF's in the ID file (QCAL). This 1) will tell the operator at a glance if one or more standards is low/high and 2) could indicate that one or two were made incorrectly (not necessarily the same thing.)

Analyze all remaining standards. Hardcopy and observe the above. Produce a Form 6 from the data system. Confirm that 1) all CCC's are below 30% and 2) the RF's for all SPCC compounds are >0.300 (Minimum RF for Chloromethane, Bromoform and 1,1,2,2-Tetrachloroethane is 0.100.) An example of an acceptable initial calibration appears in Appendix B. The BFB tune, all standard raw data and Forms 5 & 6 from LIMS are kept in a file. Each instrument has its own initial calibration.

9.5

Daily or Continuing Calibration

Continuing calibration occurs prior to analysis.

NOTE: If time remains after the initial calibration, and the 50 ppb or 10 ppb standard meets continuing calibration criteria, samples can be analyzed up to the 12 hour tune limit. The samples are quantitated against the 50 ppb or 10 ppb standard, unless client specific method requires differently.

After having satisfied BFB tune requirements, a continuing calibration standard must be analyzed. Analyze a 50 ppb or 10 ppb standard following the procedure outlined above. Confirm (CBCHK or Form 7) that all CCC's are <25% and the RF's for all SPCC's are >0.300 (Minimum RF for Chloromethane, Bromoform, and 1,1,2,2-Tetrachloroethane is 0.100.) If so, the continuing calibration is acceptable and analysis can begin.

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NOTE: If additional compounds are required, these are analyzed at the 10 or 50 ppb level only (single point calibration.)

NOTE: In most cases, all CCC's and SPCC's MUST meet requirements. Unless specified either by client-requested method or QC there may be times when analysis will proceed when continuing calibration does not meet criteria. This is approved before analysis.

The response factors are updated in the ID file. The BFB tune and continuing calibration data is kept together in a folder with all sample data analyzed within the next 12 hour period.

If continuing calibration can not be met, either new standards and/or a new 5-point calibration is needed.

9.6 Analysis

9.6.1 Method Blank

Prior to any analysis, the reagent water must be shown to be free of interferences and target compounds.

A 5 mL or 25 mL portion of reagent water is analyzed using one of the methods in Appendix A. All target compounds must be less than the quantitation limit (or <5X the quantitation limit for the following compounds: acetone, dichloro-methane, 2-butanone and toluene.)

Note: On some occasions, acetone and dichloromethane may be allowed at higher levels. IF such is the case, it is approved before analysis and documented in a case narrative.

9.6.2 Once the method blank analysis is complete and acceptable, analysis can proceed. The raw data is kept in the same folder as above.

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9.6.2 Sample Analysis9.6.2.1 Waters

Allow samples and standards to come to ambient temperature.

Remove the plunger from a 5 mL (or 25 mL) luer-lock gas-tight syringe and fill to near over-flowing. Replace the plunger, invert the syringe, and adjust the volume to 5 mLs (or 25 mLs). Confirm the absence of all air bubbles.

Draw back slightly on the plunger. Add 5 uL of the working ISS/SSS solution. Immediately add the sample to a clean purge vessel. Using the methods described in Appendix A analyze the sample. Immediately after analysis, pH check the sample. Record the pH check in the appropriate column in the instrument logbook.

Note: If lower detection limits are required, a 25 mL sample volume will be used.

If a batch is going to be analyzed, which is usually the case, load all samples following the procedure above. After the batch is loaded, replace all samples and standards back in storage.

If dilutions are to be made, the appropriate sample volume is added directly to an appropriate amount of reagent water in the 5 mL (or 25 mL) syringe. If the required amount of sample is less than 1 uL (or < 1 mL), an initial dilution is made first, and a portion of this is added to the reagent water/ISS/SSS mix in the 5 mL (or 25 mL) syringe.

Using those parameters listed in Appendix A, analyze all samples. After analysis, remove the purge vessel from the Tekmar, rinse the purge line and vessel, and place the vessel in the oven to bake at 100°C for at least an hour.

Opened sample vials are used only once unless: 1) any necessary dilutions/reruns are done the same day or 2) there are no other vials for that sample.

9.6.2.2 Soils

Before weighing any samples, check the balance using the appropriate class weights. Record the actual weights in the Balance Logbook. If a discrepancy occurs, contact quality control.

Allow samples and standards to come to ambient temperature.

Weight out 5 grams of the sample into a clean previously baked purge vessel. Record the weight to 0.1 g. Place the vessel on the Tekmar. Add reagent water to overflowing to a 5 mL syringe. Replace the plunger, invert the syringe, and with tapping, adjust to 5 mLs. Confirm there are no air bubbles. Add 5 uL of the working ISS/SSS solution. Transfer the contents to the purge vessel. Using the methods described in Appendix A, analyze the sample. Standards and samples for soil analyses are analyzed in the same manner.

If a batch of samples is to be analyzed, prepare each as above. After the batch is loaded, replace all samples and standards in storage.

If dilutions are to be made, use the following guidelines:

Dilution	Weight	Vol. MeOH Extract
1/2	2.5 g	----
1/5	1.0 g	----
1/10	0.5 g	----
1/20	5.0 g/5 mLs MeOH	250 uL
1/50	5.0 g/5 mLs MeOH	100 uL
1/100	5.0 g/5 mLs MeOH	50 uL
1/200	5.0 g/5 mLs MeOH	25 uL
1/500	5.0 g/5 mLs MeOH	10 uL
1/1000	5.0 g/5 mLs MeOH	5 uL
1/5000	5.0 g/5 mLs MeOH	1 uL

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If a larger dilution is required, either a 1/2 or 1/5 extract is made first, or an intermediate dilution is made from the 1/1 extract first. This intermediate dilution is then taken into account. The Vol. MeOH Extract above is the amount added to an appropriate amount of reagent water/ISS/SSS in the 5 mL syringe, for a 1/1 extract. This is then transferred to the purge vessel on the Tekmar. A volume of methanol is added to each blank associated with diluted soil samples.

Using those parameters in Appendix A, analyze all samples in the batch. After analysis, remove the purge vessels from the Tekmar, dispose of the contents, rinse the purge line and vessel, and place the vessel in the oven to bake at 100°C for at least one hour.

Sample vials/jars are only used once unless: 1) any dilutions/reruns are analyzed the same day or 2) there is only one jar for analysis. A portion of any MeOH extract is immediately removed from the 40 mL vial it was prepared in, and stored in a labelled 1.5 - 2.0 mL amber teflon-lined screw-capped vial kept at approximately -10°C in the dark.

The preparation and analysis is recorded in the GC/MS Volatiles Logbook, and must be completed for each days analysis. An example of a logbook page appears in Appendix C. In addition, a Tune Tracking Form is also filled out for each batch. This form will track the status of the sample once it has been analyzed. This form stays with all the data for that 12 hour period. After final review and submission for the last sample in that 12 hour batch, the quality control is removed from the system and the Tune Form is filed.

In addition, in volatiles, all samples logged into the department appear on both a hold-time summary and a Sample Tracking sheet. The Sample Tracking sheet is used in conjunction with the hold-time summary and the Tune Form to track every single sample in-house. Examples of the Tune Form and the Sample Tracking sheet appear in Appendix C.

Any sample that contains targets above the calibration range is diluted to accurately quantitate those compounds. Any sample that has shown from past experience to be high in background/targets, or appears to be so, is first screened by GC/FID. The intensity of the signal of the non-internal standard peaks is compared to that of the internal standards to give a reasonable estimation of an appropriate dilution.

9.6.2.3 Drum/Waste Samples

- Non-methanol Miscible

These are treated as soil samples. Extracts are prepared as above.

- Methanol Miscible

A dilution is made directly into methanol. A portion of this is then added to the reagent water/ISS/SSS mixture in the 5 mL syringe. The initial dilution is taken into account when calculating the final dilution factor. A volume of methanol is added to each blank associated with diluted drum samples.

10.0 CALCULATIONS

10.1 Computer Data Production/Reduction

The RTE/Data System produces a Total Ion Chromatogram (TIC), header, quant report and background subtracted spectra. For those clients requiring it, a 10 tentatively identified compound search is also performed. The data system will produce an integration listing and tentative identification of each hit found at the selected percentage of the largest peak present. All data for each sample is kept together. All data for all samples run during that 12 hour period remains together until operator data workup begins. At which time all appropriate QC is photocopied for each RFW# batch in that run batch.

10.1.1 Quantitation of Target Compounds

Quantitation of the target compounds is performed by the data system and LIMS but can be accomplished as follows:

WATERS:

$$\text{concentration (ug/L)} = \frac{[A_x \times I_c]}{[A_{is} \times RF]} \times DF$$

Where:

A_x = area of characteristic ion for target
 I_s = concentration of internal standard (ng)
 A_{is} = area of characteristic ion for int. std.
 RF = response factor for target
 DF = dilution factor (if any)

SOILS:

$$\text{concentration (ug/kg)} = \frac{[A_x \times I_s]}{[A_{is} \times RF \times D]} \times DF$$

Where:

All variables are equal and

D = (100 - % moisture in sample/100) or 1 for wet weight. (As in the case of drum samples)

The sample volume is considered to be "constant" for calculation purposes, and is always entered as five (5) in the Lotus Spreadsheet. Less sample volume (in the case of waters) and extract volume (in the case of soils/drums) are taken into account in the dilution factor at LIMS.

10.1.2 Quantitation of TIC's (Tentatively Identified Compounds)

Quantitation of TIC's is performed by the RTE Data system, operator and LIMS. The formulas above for waters and soils can be used with the following modifications. A_x and A_{is} should be taken from the total ion integration listing accompanying the TIC report produced by the data system. The nearest non-interfered with internal standard should be used. The RF is assumed to be one (1). The concentration is therefore an estimate and is flagged as such with a "J". Any TIC also found in the method blank is flagged with a "JB". With the present LIMS system, the calculations are much simpler.

$$\text{Total ng of TIC} = \frac{\text{**area of TIC} \times \text{ng of its ISTD (50)}}{\text{area of that ISTD}}$$

** The TIC program (*TRQAL or SMLTIC) has been modified to draw the integrated chromatogram in two portions. The operator should visually confirm that the integration is correct. If not, the peak in question must be manually integrated. The total ng of TIC is entered into a spreadsheet at LIMS which then takes into account total solids and the dilution factor in the final calculation of the TIC concentration.

10.2 Operator Data Reduction/Review

The operator reviews the data produced by the RTE system and 1) makes judgements concerning the "realness" of those target compounds found and 2) makes judgements concerning the identification of the tentatively identified compounds and 3) modifies the output to produce a data package reflective of those decisions.

10.2.1 Initial Review

Operator determines that the analysis in itself is acceptable. This means:

QC Samples/Criteria are met

- The method blank contains no interferences or target compounds at the EQL or 5X the EQL for previously listed compounds.
- ALL surrogates are in control in the blank
- ALL surrogates in samples are in control (current surrogate limits are listed below)
- LCS recoveries are advisory only as in-house data is being generated, and will be used to set limits (current limits are listed below)

Water Surrogate Limits: 80 - 120%

Soil Surrogate Limits: Refer to SW-846 Method 8240

Water Matrix Spike Limits: 80 - 120%

Soil Matrix Spike Limits: Refer to SW-846 Method 8240

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The sample does not require any further dilutions or analysis at a more concentrated level. Visually confirm complete integration for any large and/or saturated target compounds. Dilutions are made to keep the target in the upper half of the calibration range. The MS and MSD are never diluted to get spiked or non-spiked compounds within range, as this would reduce the matrix affect assessment.

The sample does not require re-analysis for any other reason (i.e. leak, analysis past tune time, ISTD areas low, etc...).

10.2.2 Identification of Targets

The following guidelines are used in the positive identification of target compounds.

"elution of component at the same relative retention time as the standard component."

The elution times should compare within 0.06 min. The standard must be run on the same 12 hour period as the sample. If co-eluting analytes interfere with the comparisons of retention times, other ions characteristic to that compound can be used to confirm relative retention times.

10.2.3 "correspondence of the sample component and standard component mass spectrum." Comparisons of sample spectra to standard spectra must be made using standard spectra obtained from the GC/MS system.

All ions present in the standard spectrum at a 10% relative intensity (most abundant ion being 100%) **MUST** be present in the sample.

The relative intensities of the above ions must agree within $\pm 20\%$, between the standard and sample. If an ion is 50% intensity in the standard the corresponding ion must be between 30 and 70% in the sample.

Ions $> 10\%$ in the sample but not present in the standard must be considered and accounted for. (A user program exists to aid in this).

Operator judgement. If a compound can not be verified by the above, but in the operators technical judgement the ID is correct, it is reported as such.

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10.2.4 Once all positive identification is made, the output file is modified to reflect these decisions (the integrity of the original file is not destroyed). In each case where the file has been edited or manual integration has taken place, the operator must identify, initial and date the changes. An ascii file is created for later transfer to LIMS. All analytes at concentrations less than the Estimated Quantitation Limit are automatically flagged with a J.

10.2.5 Identification of TIC's

In general, up to as many as 10 non-target compounds are tentatively identified by the data system and operator. Compounds with responses < 10% of the nearest ISTD are not identified. The data system provides the operator with a SUB ADC C sample spectrum, spectra of the first three matches and a listing of two other possibilities. Molecular formulas, molecular weights and CAS #'s are included. The following guidelines are used:

10.2.6 Relative intensities of major ions in the reference spectrum should be present in the sample (ions > 10%).

- Relative ions should agree within $\pm 20\%$.
- Molecular ions in the reference should be in the samples.
- Review the possibility of background and/or co-eluting compounds for those ions present in the sample but not in the standard.
- If ions are present in the sample but not in the standard, review the possibility of the presence of background or co-eluting compounds.
- If ions are present in the standard but not in the sample, review the possibility that the ions were subtracted out because they are also common to the background or co-eluting compounds.
- In the event no valid interpretation can be made, the compound is called "unknown".

Interpretation can be often narrowed down to a class of compounds, molecular formula or weight.

10.3 Final Review

Once 1) the analysis is determined to be acceptable and 2) the initial review and data reduction has occurred (verifiable on the Tune Form) 3) the results transferred to LIMS as ascii files and 4) all forms are produced, the package is put together and submitted for its first 100% review. The checklist in Appendix D is used as an aid to review.

In actuality, some of the forms will not print correctly or at all if many of the review checks are not correct. Some of the checks are performed even as the operator is working up the data to the level accepted and used by LIMS. In this manner, the data is being reviewed as soon as it is produced by the data system.

Upon the first 100% review, the review form is initialed and dated as reviewed. The package, with its review sheet, comments and any C/A forms is submitted to the unit leader or section manager for a second review and validation. Once the data passes review and report generation 1) archival is verified or completed at that time (verification available from the VOA archive logbook) or later and 2) the files are purged from the data system. A hardcopy of the purge file is obtained, initialed and dated and serves as documentation.

The complete analysis scheme can be summarized below (Section 11.1 & 11.2) and in Appendix E. The entire sample tracking system can be summarized in Appendix E.

11.0 QUALITY CONTROL

Quality Control is accomplished through 1) daily tuning and calibration checks and 2) preparation QC traceable through individual extraction batches.

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11.1 Initial Calibration

PFTBA			
BFB TUNE		Prior to 5-Point Curve	*control chart
200 or 20 ng \			
150 or 15 ng			
100 or 10 ng	5-Point	Need dependent on	*control chart
50 or 5 ng		situation. See	
10 or 1 ng /		Section 9.1	

11.2 Daily Analysis

PFTBA		
BFB	Prior to continuing calibration	*control chart
Daily Calibration Standard	Prior to samples	*control chart

Samples *

*Any given 12 hour period contains a tune, standard, blank and blank spike. Preparation QC is at a 5% frequency. Instrumental controls are outlined above and further discussed in the procedure section.

<u>Prep QC</u>	<u>Frequency</u>
Blank	Prior to analysis
LCS	1 per analysis batch
MS/MSD's	at least 1 set in 20
Surrogates	every blank, sample and QC Sample
QC Charting	1 set per month

11.3 The department will review the quality controls as follows:

11.3.1 At least one method blank and at least one Lab Control Standard (LCS) will be included in each laboratory batch. Regardless of the matrix being processed, the LCS and method blanks will be in an aqueous media.

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- 11.3.2 The method blanks will be examined to determine if contamination is being introduced in the laboratory.
- 11.3.3 The LCS will be examined to determine accuracy and precision.
- 11.3.4 Accuracy will be measured by the percent recovery (%R) of the LCS. The recovery must be in range, as determined by statistical analysis, in order to be considered acceptable. Additionally, %R will be plotted on control charts to monitor method accuracy. (Current limits listed in Section 10.2.1.)
- 11.3.5 Precision will be measured by the reproducibility of both blank spikes and will be calculated as Relative Percent Difference (RPD). Results should be <20%. (Control Limits are generated on a quarterly basis).
- 11.3.6 Surrogate Compounds will be added to every sample to measure performance of the analysis. Results must agree within statistical control limits in order to be considered acceptable. (Current limits listed in Section 10.2.1.)

12.0 **CORRECTIVE ACTIONS**

Listed below are the steps to be taken when an out-of-control situation occurs. The analyst **MUST**:

- demonstrate that all of the problems creating the out-of-control situation were addressed;
- document the problem and the action that was taken to correct the problem;
- document that an in-control situation has been achieved; and
- receive approval (signature) of the unit leader, section manager or QC personnel prior to release of data associated with the problem.

Discussed below are the suggested and required courses of action when an out-of-control situation has occurred.

All surrogate recoveries are calculated.

If ANY surrogates are outside limits in the Method Blank, it must be re-analyzed. Analyses CAN NOT proceed until an in-control situation is demonstrated. Re-analyze the blank. If surrogates are still out, the instrument may need to be re-tuned (BFB) and/or another calibration standard analyzed. If the problem persists, further maintenance action may be required (i.e. trap replacement, clean separator).

Before pursuing other measures, check to be sure that:

- calculations are correct
- concentrations of the surrogates in the spiking solution are correct
- the correct amount of ISS/SSS solution was added
- ISS/SSS areas are reasonable

If any surrogates in a sample are outside limits, check the above first. Any sample that has a surrogate out must be re-analyzed. The re-analysis can take the form of a dilution. If the surrogate (s) is/are still outside limits a matrix effect is demonstrated and both reports are submitted.

If all surrogates are in-control on the re-analysis, only the second analysis is reported.

Every effort is made to complete the re-analysis within hold-time. If this is impossible (i.e. capacity hold-times preclude re-analyses hold-time) both reports may be submitted. This is documented in the narrative.

If the sample with the out-of-control surrogates is the same sample on which the MS and MSD has been performed, and the pattern is duplicated, then re-analysis is NOT required. Documentation of the similarities is required.

MS and MSD recoveries and RPD's are advisory only. However, if problems are frequent, investigation of the system is necessary.

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12.1 BFB Criteria

If BFB criteria can not be met, determine if the source of the problem is instrumental or tune related. Inspect overall sensitivity, possible excessive background, the proportionality of the masses, relative abundances of the target masses. If it seems tune related, adjust the tune parameters in Manual Tune slightly, until acceptance is achieved. If the problem seems instrumental, perform suggested trouble-shooting to locate and correct the problem (Suggestions can be found in most of the manuals). NO analysis can proceed until criteria are met. Each instrument will have its own idiosyncracies.

12.2 Initial Calibration

If initial calibration can not be met, determine if the problem is analytical or instrumental. Some suggested questions to ask would be:

- were the standards prepared correctly?
- was the proper amount analyzed?
- check the chromatogram - did something happen on one or two analyses; i.e. a leak
- check the response factors - is one concentration level very high or low? re-analyze it
- how old are the standards?

The course of action depends on the problem

12.3 Continuing Calibration

If continuing calibration can not be met, determine if the problem is analytical or instrumental. Some suggestions:

- check the chromatography
 - is overall sensitivity low?
 - excessive background?
 - how old is the standard?
 - need a new 5-point?
- has the tune wandered?

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Compare the relative abundances of 69, 131 and 219 from that days manual tune to those on the day the initial calibration was analyzed. Slight adjustments to the tune may bring the standard in.

Certain compounds will help indicate what the problem is. The course of action depends on the problem.

12.4 Blank

If the method blank is/appears to be contaminated:

- re-analyze it on a different position

If contamination is still present, the problem may be in one of the common elements, such as the trap, transfer line, port valve or column or the internal standard solution. Baking the trap/column and running position blanks may be necessary. If contamination has occurred beyond that, and maintenance is required (i.e. replace trap) it is documented in the Maintenance Logbook. Analysis CAN NOT proceed until the blank is free of contamination.

12.5 LCS's

Limits must be generated by in-house data. Advisory only. Outliers are documented.

12.6 Matrix Spikes

MS and MSD recoveries and RPD's are advisory only and function to illustrate matrix effects on the efficiency and completeness of target analyses. Outliers are documented.

Maintenance logbooks do exist. Major maintenance and problems are noted in these logbooks. The entry number is transferred to the appropriate run log, initialed and dated. Other courses of action taken for out-of-control situations are documented either on the review form or the case narrative or in the operator logbook itself.



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13.0 **HEALTH AND SAFETY**

As always, general laboratory safety practices should always be followed. Waste samples should be handled with care due to the uncertainty of the properties and contents involved.

Refer to the specific MSDS for the hazardous properties of ANY chemical or reagent involved in this procedure.

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Table 1

Characteristic Mass (m/z) for Purgeable Organic Compounds

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Benzene	78	77, 158
Bromobenzene	156	48, 130
Bromochloromethane	128	85, 127
Bromodichloromethane	83	175, 254
Bromoform	173	96
Bromomethane	94	92, 134
n-Butylbenzene	91	134
sec-Butylbenzene	105	91, 134
tert-Butylbenzene	119	119
Carbon tetrachloride	117	77, 114
Chlorobenzene	112	66
Chloroethane	64	85
Chloroform	83	52
Chloromethane	50	126
2-Chlorotoluene*	91	126
4-Chlorotoluene*	91	155, 157
1,2-Dibromo-3-chloropropane	75	127
Dibromochloromethane	129	109, 188
1,2-Dibromoethane	107	95, 174
Dibromomethane	93	111, 148
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	87
Dichlorodifluoromethane	85	65, 83
1,1-Dichloroethane	63	98
1,2-Dichloroethane	62	61, 63
1,1-Dichloroethene	96	61, 98
cis-1,2-Dichloroethene	96	61, 98
trans-1,2-Dichloroethene	96	112
1,2-Dichloropropane	63	78
1,3-Dichloropropane	76	97
2,2-Dichloropropane	77	110, 77
1,1-Dichloropropene	75	106
Ethylbenzene*	91	223, 227
Hexachlorobutadiene	225	120
Isopropylbenzene	105	134, 91
p-Isopropylbenzene	119	86, 49

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Table 1
 (continued)
Characteristic Mass (m/z) for Purgeable Organic Compounds

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Methylene chloride	84	86, 49
Naphthalene	128	---
n-Propylbenzene*	91	120
Styrene	104	78
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	166	168, 129
Toluene*	92	91
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1-Trichloroethane*	97	99, 61
1,1,2-Trichloroethane*	83	97, 85
Trichloroethene	95	130, 132
Trichlorofluoromethane	101	103
1,2,3-Trichloropropane*	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl chloride	62	64
o-Xylene*	106	91
m-Xylene*	106	91
p-Xylene*	106	91
<u>Internal Std./Surrogates</u>		
4-Bromofluorobenzene	95	174, 176
1,2-Dichlorobenzene-d ₄	152	150
Pentafluorobenzene	168	
Chlorobenzene-d ₅	117	
1,4-Difluorobenzene	114	
1,2-Dichloroethane-d ₄	65	
Toluene-d ₈	98	

*Note: The primary and secondary ions listed here are taken directly from SW-846 Method 8260. There are instances where we have chosen different ions (i.e. interferences from co-eluting compounds or for enhances identification purposes.)

Table 2

Estimated Quantitation Limits for Volatile Analytes^a

	Estimated Quantitation Limits		
	Ground Water (ug/L)		Low Soil/Sediment ^b (ug/kg)
Volume of water purged	5 mL	25 mL	
All analytes in Table 1	5	1	5

^aEstimated Quantitation Limit (EQL) - The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5 to 10 times the MDL. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes the EQL analyte concentration is selected for the lowest non-zero standard in the calibration curve. Sample EQLs are highly matrix-dependent. The EQLs listed herein are provided for guidance and may not always be achievable. See the following information for further guidance on matrix-dependent EQLs.

^bEQLs listed for soil/sediment are based on wet weight. Normally data is reported on a dry weight basis; therefore, EQLs will be higher, based on the percent dry weight in each sample.



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Figure 1

Example of Maintenance Log

WESTON-Gulf Coast, Inc.
GC/MS Volatile Maintenance Log
Instrument 3

Date of Maintenance: _____	Entry No.: _____
Analyst: _____	
Description: _____	

Follow Up: _____	

Analyst: _____	Date: _____

Date of Maintenance: _____	Entry No.: _____
Analyst: _____	
Description: _____	

Follow Up: _____	

Analyst: _____	Date: _____

Date of Maintenance: _____	Entry No.: _____
Analyst: _____	
Description: _____	

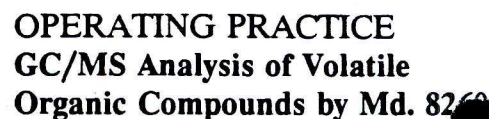
Follow Up: _____	

Analyst: _____	Date: _____

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Figure 2

NEAT GC/MS STANDARDS LOGBOOK					
<u>Neat Standards Ref.#</u>	<u>Lot#</u>	<u>Compound/Kit</u>	<u>Rec'd</u>	<u>Opened</u>	<u>Comments</u>
<p>FIGURE 2. Neat Standards Logbook: This logbook is used to record/track ALL neat standards, either as single compounds or kits, received and used by the GC/MS Department. Neat standard reference numbers are coded by a single letter designating the compound type followed by the date received.</p> <p>S = Surrogate T = Target I = Internal Standard M = Matrix Spike</p> <p>All neat standards are recorded and marked with a code upon receipt, then stored at -10°C until use.</p>					

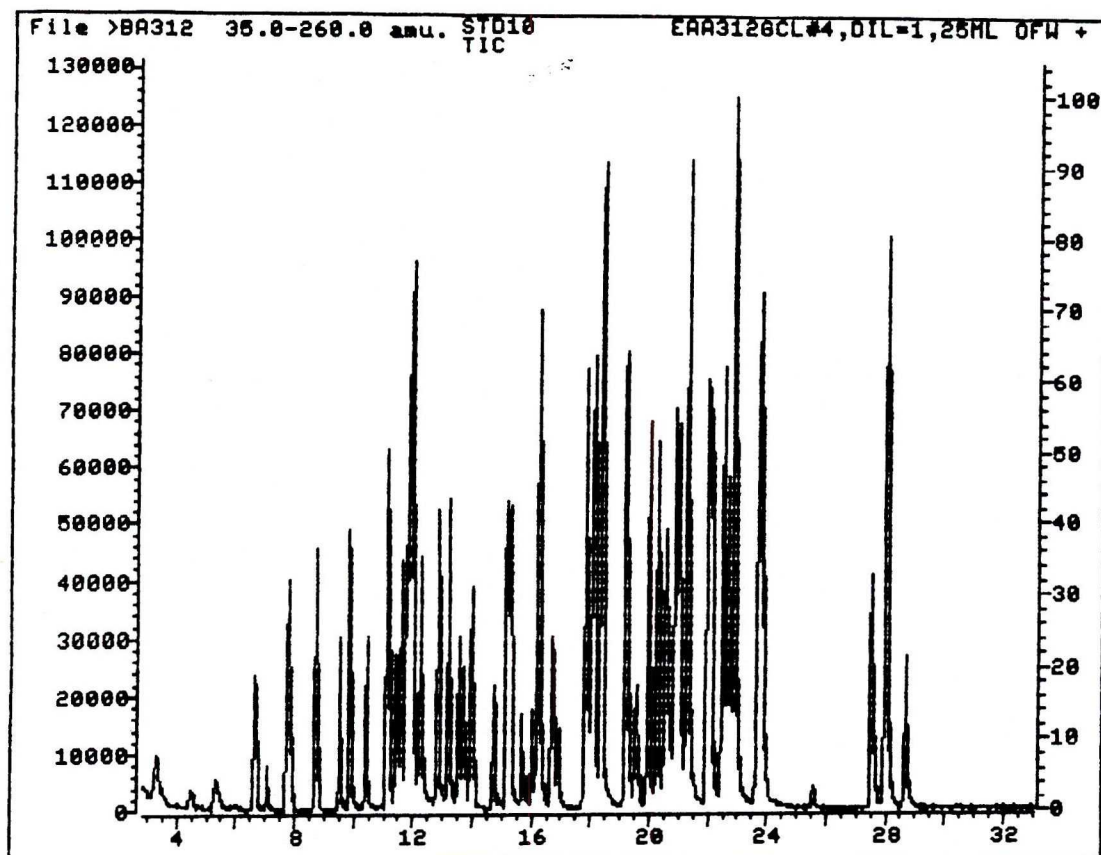


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Figure 4

Total Ion Chromatogram for Normal 25 mL Purge Water

Figure 4

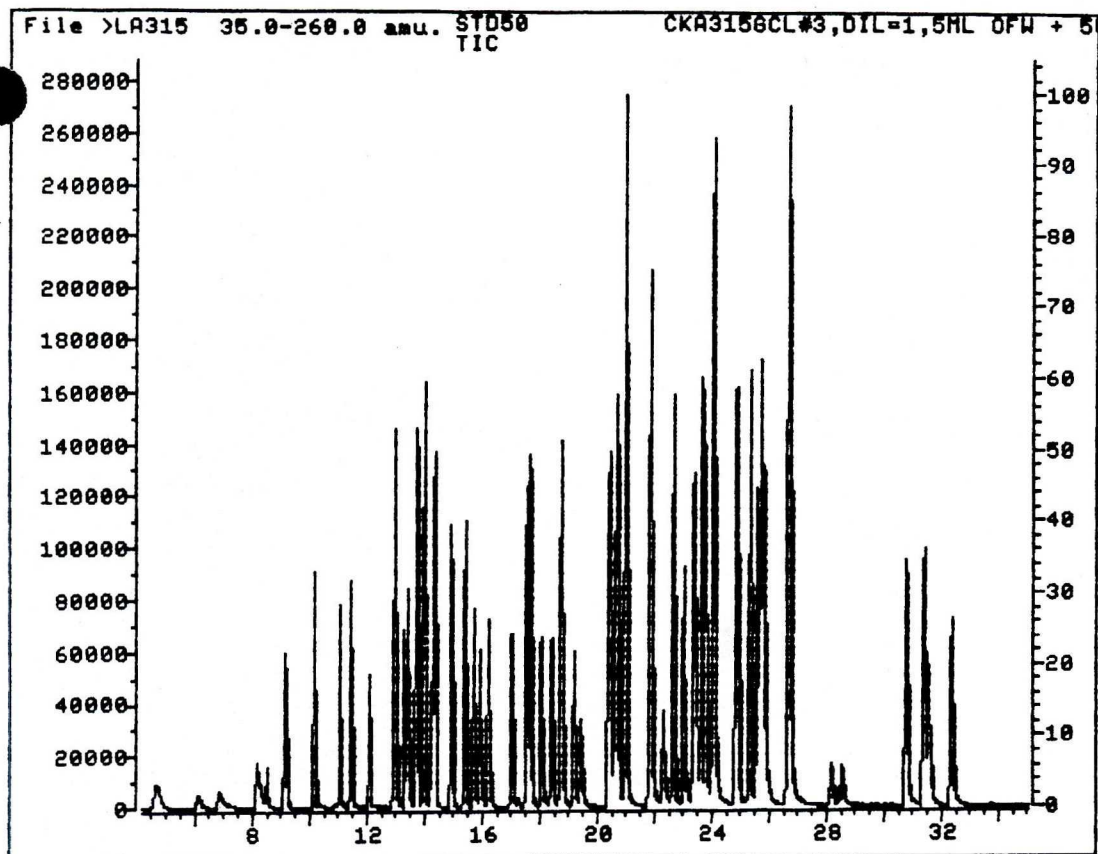


TOTAL ION CHROMATOGRAM FOR NORMAL 25ml PURGE H₂O

MS data file header from : >BA312

Sample: STD10 EAA312 Operator: MANAGER MS 3/12/93 14:51
 Misc : GCL#4,DIL=1,25ML OFW + 5UL ISS 545 + 10UL 543
 Sys. #: 4 MS model: 70 SW/HW rev.: IA ALS #: 0
 Method file: WESTON Tuning file: TUNE4 No. of extra records: 2
 Source temp.: 0 Analyzer temp.: 250 Transfer line temp.: 0

Chromatographic temperatures :	-20.	50.	170.	0.	0.
Chromatographic times, min. :	4.0	0.0	17.0	0.0	0.0
Chromatographic rate, deg/min:	15.0	6.0	0.0	.5	0.0



TOTAL ION CHROMATOGRAM FOR NORMAL 56 SOILS

MS data file header from : >LA315

Sample: STD50 CKA315 Operator: MANAGER MS 3/15/93 11:21
Misc : GCL#3,DIL=1,5ML OFW + 5UL ISS 545 + 2.5UL 559
Sys. #: 3 MS model: 96 SW/HW rev.: IA ALS #: 0
Method file: JOHN Tuning file: TUNE3 No. of extra records: 2
Source temp.: 150 Analyzer temp.: 180 Transfer line temp.: 250

Chromatographic temperatures :	-20.	50.	170.	0.	0.
Chromatographic times, min. :	4.0	0.0	17.0	0.0	0.0
Chromatographic rate, deg/min:	15.0	6.0	0.0	0.0	0.0



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APPENDIX A.

Method Listings

Tekmar Conditions

Flow Settings

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METHOD FILE LIST

Method File: DON

GC TYPE: 5996
Column: Cap

Run Type: SCAN, GC, EI
Splitless: Yes

Temperature:	Inj. P	Xfer L	Source	Analyzer	FID
	160.0	250.0	150.0	180.0	.0

GC/DIP	LEVEL 1	LEVEL 2	LEVEL 3	LEVEL 4	POST RUN
Temp 1	20.0	160.0	0.0	0.0	0.0
Time 1	2.0	5.0	0.0	0.0	0.0
Rate	10.0	0.0	0.0	0.0	
Temp 2	160.0	0.0	0.0	0.0	
Time 2	5.0	0.0	0.0	0.0	

Run Time: 21.00
Scan Start Time: 6.00
Splitless valve time: 0.00

	ON	OFF	ON	OFF
Solvent Diverter Valve:	327.0	327.0	327.0	327.0
Relay #1:	0.10	0.30	327.0	327.0
Relay #2:	0.10	0.30	327.0	327.0
Triac #0:	327.0	327.0	327.0	327.0
Triac #1:	327.0	327.0	327.0	327.0

ALS Operating Conditions

Number of sample washes: 2
Solvent A Washes: 2
Sample Viscosity Wait: 0

Number of Sample Pumps: 6
Solvent B Washes: 0
Injection Mode is FAST

Scan Parameters: Mass Range: 35 - 260
 Multiplier Voltage: 1984
 Number of A/D Samples: 8
 GC Peak Threshold: 10000 counts
 Threshold: 15 counts



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OPERATING PRACTICE
GC/MS Analysis of Volatile
Organic Compounds by Md. 8

Eff. Date: 04/26/93 Initiated By: QC Department Approved By: J.A. Kaczinski Authorized By: A. M. Henry SP No. 21-16G-8260

METHOD FILE LIST

Method File: ROYF GC TYPE: 5890 Run Type: SCAN, GC, EI
Column: Cap Splitless: Yes

Temperature: Inj. P Xfer L Source
160.0 250.0 0.0

GC/DIP		LEVEL A	LEVEL B	POST RUN
Temp 1	100.0	200.0	0.0	0.0
Time 1	2.0	5.0	0.0	0.0
Rate	5.0	0.0	0.0	
Temp 2	200.0	0.0	0.0	
Time 2	5.0	0.0	0.0	

Oven Equilibration Time: 0.00 min

Run Time: 15.00
Scan Start Time: 7.00
Splitless valve time: 0.00

	ON	OFF	ON	OFF
Relay #1:	0.10	0.30	327.0	327.0
Relay #2:	0.10	0.30	327.0	327.0
Triac #0:	327.0	327.0	327.0	327.0
Triac #1:	327.0	327.0	327.0	327.0

Scan Parameters: Mass Range: 35 - 260
Multiplier Voltage: 1606
Number of A/D Samples: 16
GC Peak Threshold: 10000 counts
Threshold: 10 counts



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OPERATING PRACTICE
**GC/MS Analysis of Volatile
Organic Compounds by Md. 8260**

Eff. Date: 04/26/93 Initiated By: QC Department Approved By: J.A. Kaczinski Authorized By: A. M. Henry SP No. 21-16G-8260

METHOD FILE LIST

Method File: JOHN GC TYPE: 5996 Run Type: SCAN, GC, EI
Column: Cap Splitless: Yes

Temperature:	Inj. P	Xfer L	Source	Analyzer	FID
	160.0	250.0	150.0	180.0	.0

GC/DIP	LEVEL 1	LEVEL 2	LEVEL 3	LEVEL 4	POST RUN
Temp 1	0.0	80.0	170.0	0.0	170.0
Time 1	4.0	2.0	10.0	0.0	3.0
Rate	10.0	4.0	0.0	0.0	
Temp 2	80.0	170.0	0.0	0.0	
Time 2	2.0	10.0	0.0	0.0	

Run Time: 28.50
Scan Start Time: 1.50
Splitless valve time: 0.00

	ON	OFF	ON	OFF
Solvent Diverter Valve:	327.0	327.0	327.0	327.0
Relay #1:	0.10	0.30	327.0	327.0
Relay #2:	0.10	0.30	327.0	327.0
Triac #0:	327.0	327.0	327.0	327.0
Triac #1:	327.0	327.0	327.0	327.0

ALS Operating Conditions

Number of sample washes: 2	Number of Sample Pumps: 6
Solvent A Washes: 2	Solvent B Washes: 0
Sample Viscosity Wait: 0	Injection Mode is FAST

Scan Parameters: Mass Range: 35 - 260
Multiplier Voltage: 1889
Number of A/D Samples: 8
GC Peak Threshold: 10000 counts
Threshold: 20 counts



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OPERATING PRACTICE
GC/MS Analysis of Volatile
Organic Compounds by Md. 82

Eff. Date: 04/26/93 Initiated By: QC Department Approved By: J.A. Kaczinski Authorized By: A. M. Henry SP No. 21-16G-8260

METHOD FILE LIST

Method File: WESTON GC TYPE: 5890 Run Type: SCAN, GC, EI
Column: Cap Splitless: Yes

Temperature: Inj. P Xfer L Source
 160.0 250.0 0.0

GC/DIP		LEVEL A	LEVEL B	POST RUN
Temp 1	-20.0	80.0	170.0	0.0
Time 1	4.0	2.0	10.0	0.0
Rate	10.0	4.0	0.0	
Temp 2	80.0	170.0	0.0	
Time 2	2.0	10.0	0.0	

Oven Equilibration Time: 0.50 min

Run Time: 37.00

Scan Start Time: 2.50

Splitless valve time: 0.00

	ON	OFF	ON	OFF
Relay #1:	0.10	0.30	327.0	327.0
Relay #2:	0.10	0.30	327.0	327.0
Triac #0:	327.0	327.0	327.0	327.0
Triac #1:	327.0	327.0	327.0	327.0

Scan Parameters: Mass Range: 35 - 260
 Multiplier Voltage: 1748
 Number of A/D Samples: 16
 GC Peak Threshold: 10000 counts
 Threshold: 10 counts

Eff. Date: 04/26/93 Initiated By: QC Department Approved By: J.A. Kaczinski Authorized By: A. M. Henry SP No. 21-16G-8260

Tekmar Conditions

Trap Temp. Prior to Purge	< 30
Desorb Preheat	100
Desorb	180
Bake	225
Purge Time	10 - 11 min
Desorb	4 min
Bake Time	10 - 11 min

The bake time on instrument #6 is slightly longer

Flow Conditions

Purge Pressure	20 psi
Purge Flow Rate	40 mLs/min

Flow Adjustment

Capillary Column: 5970/5971/MSD's;
5995 Benchtop

- Make-up gas off/separator pump on: flow through separator is 5-10 mLs/minutes.

- Open make-up gas: adjust until you achieve ~30 mLs/minute through the separator. (On MSD's - adjust to 0.5 torr on gauge)

(Flow into the Mass Spec is ≤ 1 mL/minute)

Approximate Vacuums

5971	$\sim 5 \times 10^{-6}$ torr
5970	$\sim 2-3 \times 10^{-5}$ torr
5995	$\sim 1-2 \times 10^{-5}$ torr



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GC/MS Analysis of Volatile
Organic Compounds by Md. 8

Eff. Date: 04/26/93 Initiated By: QC Department Approved By: J.A. Kaczinski Authorized By: A. M. Henry SP No. 21-16G-8260

APPENDIX B.

ID File Listings

Initial Calibration Example

Quant ID File: ID_826::U2

Title: ID FILE FOR CAPILLARY METHOD 8260 INST. #4 WATERS

Last EDIT Date: 930313 10:58

Last Calib Date: 930323 11:46

RT Window (+/- min): 1.00
Max Hits/Compound: 5
Minimum Area: 500
Peak/Base Peak Ratio: 40.00 %
Slope Sensitivity: .200
Subtraction Method: 3
Ignore max check: NO
Auto Qdel Method: 5
Units of Conc: ppb

Number of Compounds: 82

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
1)	*Pentafluorobenzene	(I)	11.78(1.000)	1.00000	10.00
2)	Dichlorodifluoromethane	(T)	3.17(.269)	3.31883	10.00
3)	Chloromethane	(T)	4.38(.372)	.90686	10.00
4)	Vinyl Chloride	(T)	5.30(.450)	1.39560	10.00
5)	Bromomethane	(T)	6.57(.558)	2.23762	10.00
6)	Chloroethane	(T)	6.97(.591)	.80301	10.00
7)	Trichlorofluoromethane	(T)	7.69(.653)	5.42562	10.00
8)	1,1-Dichloroethene	(T)	8.63(.732)	1.88998	10.00
9)	Methylene Chloride	(T)	9.41(.799)	1.37767	10.00
10)	Acetone	(T)	10.47(.889)	.48637	10.00
11)	Acrolein	(T)	8.59(.729)	.00014	1600.00
12)	Acrylonitrile	(T)	9.79(.831)	.04288	320.00
13)	trans-1,2-Dichloroethene	(T)	9.76(.829)	1.93441	10.00
14)	1,1-Dichloroethane	(T)	10.31(.875)	3.07527	10.00
15)	2,2-Dichloropropane	(T)	11.04(.937)	2.28096	10.00
16)	cis-1,2-Dichloroethene	(T)	11.06(.938)	1.74699	10.00
17)	Chloroform	(T)	11.49(.975)	3.99303	10.00
18)	Bromochloromethane	(T)	11.35(.963)	.89470	10.00
19)	1,1,1-Trichloroethane	(T)	11.68(.992)	3.71881	10.00
20)	2-Butanone	(T)	11.12(.944)	.13639	10.00
21)	Carbon Disulfide	(T)	8.80(.747)	3.82985	10.00
22)	Iodomethane	(T)	8.78(.745)	8.84562	20.00
23)	Vinyl Acetate	(T)	10.47(.889)	.48229	10.00
24)	*1,4-Difluorobenzene	(I)	12.74(1.000)	1.00000	10.00
25)	Carbon Tetrachloride	(T)	11.88(.932)	.89246	10.00
26)	1,2-Dichloroethane-d4	(S)	12.11(.951)	.22629	10.00
27)	1,1-Dichloropropene	(T)	11.90(.934)	.51409	10.00
28)	Methyl-tert-Butyl Ether	(T)	9.83(.772)	0.00000	10.00
29)	Benzene	(T)	12.17(.955)	.73344	10.00
30)	1,2-Dichloroethane	(T)	12.21(.958)	.23227	10.00
31)	Toluene-d8	(S)	15.05(1.181)	.96588	10.00
32)	Trichloroethene	(T)	13.09(1.028)	.48837	10.00
33)	2-Chloroethyl vinyl ether	(T)	14.41(1.131)	.06056	10.00
34)	1,2-Dichloropropane	(T)	13.44(1.055)	.28915	10.00
35)	Bromodichloromethane	(T)	13.90(1.091)	.71356	10.00
36)	Dibromomethane	(T)	13.62(1.069)	.25532	10.00
37)	4-Methyl-2-Pentanone	(T)	14.93(1.172)	.07122	10.00
38)	trans-1,3-Dichloropropene	(T)	15.62(1.226)	1.27202	10.00
39)	Toluene	(T)	15.17(1.191)	1.01335	10.00

40)	1,2-Dibromoethane	(T)	16.79(1.318)	.29742	10.00
41)	cis-1,3-Dichloropropene	(T)	14.62(1.148)	.34242	10.00
42)	1,1,2-Trichloroethane	(T)	15.91(1.249)	.16938	10.00
43)	*Chlorobenzene-d5	(I)	17.68(1.000)	1.00000	10.00
44)	Tetrahydrofuran	(T)	15.03(.850)	0.00000	10.00
45)	Bis(Chloromethyl)Ether	(T)	13.89(.786)	0.00000	6400.00
46)	2-Hexanone	(T)	16.43(.929)	.07863	10.00
47)	Ethyl methacrylate	(T)	15.80(.894)	.19894	20.00
48)	Tetrachloroethene	(T)	16.10(.910)	1.05323	10.00
49)	1,3-Dichloropropane	(T)	16.18(.915)	.31498	10.00
50)	Dibromochloromethane	(T)	16.59(.938)	.64263	10.00
51)	Chlorobenzene	(T)	17.74(1.003)	1.00923	10.00
52)	1,1,1,2-Tetrachloroethane	(T)	17.92(1.013)	.61154	10.00
53)	Ethylbenzene	(T)	18.00(1.018)	.49399	10.00
54)	p-Xylene	(T)	18.23(1.031)	2.93726	10.00
55)	m-Xylene	(T)	18.23(1.031)	2.93726	10.00
56)	o-Xylene	(T)	19.03(1.076)	1.45111	10.00
57)	Styrene	(T)	19.07(1.079)	.82965	10.00
58)	Bromoform	(T)	19.41(1.097)	.37937	10.00
59)	*1,2-Dichlorobenzene-d4	(I)	23.55(1.000)	1.00000	10.00
60)	Bromofluorobenzene	(S)	20.14(.855)	.97337	10.00
61)	Isopropylbenzene	(T)	19.85(.843)	2.30424	10.00
62)	1,1,2,2-Tetrachloroethane	(T)	20.55(.873)	.31823	10.00
63)	Bromobenzene	(T)	20.42(.867)	.70202	10.00
64)	1,2,3-Trichloropropane	(T)	20.59(.874)	.08397	10.00
65)	n-Propylbenzene	(T)	20.75(.881)	.54787	10.00
66)	2-Chlorotoluene	(T)	20.87(.886)	.53287	10.00
67)	trans-1,4-dichloro-2-butene	(T)	20.60(.875)	.31496	20.00
68)	4-Chlorotoluene	(T)	21.12(.897)	.56787	10.00
69)	1,3,5-Trimethylbenzene	(T)	21.16(.899)	1.91364	10.00
70)	tert-Butylbenzene	(T)	21.86(.929)	1.97576	10.00
71)	1,2,4-Trimethylbenzene	(T)	21.98(.934)	1.80746	10.00
72)	sec-Butylbenzene	(T)	22.37(.950)	2.77682	10.00
73)	p-Isopropyltoluene	(T)	22.73(.965)	2.29034	10.00
74)	1,3-Dichlorobenzene	(T)	22.53(.957)	1.13387	10.00
75)	1,4-Dichlorobenzene	(T)	22.75(.966)	1.09543	10.00
76)	1,2-Dichlorobenzene	(T)	23.59(1.002)	.99663	10.00
77)	n-Butylbenzene	(T)	23.67(1.005)	2.30917	10.00
78)	1,2-Dibromo-3-Chloropropane	(T)	25.45(1.081)	.04389	10.00
79)	1,2,4-Trichlorobenzene	(T)	27.38(1.163)	.76701	10.00
80)	Hexachlorobutadiene	(T)	27.85(1.183)	1.44748	10.00
81)	Naphthalene	(T)	27.93(1.186)	.40273	10.00
82)	1,2,3-Trichlorobenzene	(T)	28.52(1.211)	.53017	10.00

* Compound is ISTD